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Effect of Heating Temperature on Wear Rate, Tensile Strength, and Crystallinity of Cantula Fiber-Reinforced Magnesium/Hydroxyapatite/ Shellac for Bone Screw Material

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Abstract
Bone screws are screws for bone that are joined to support plates. Bone screws generally use metal as the primary material because of its high mechanical properties, such as stainless steel and titanium. Currently, many biomaterials for bone screws are being developed, which can be degraded by the body so that there is no need for surgical removal of bone plates and screws. This study aimed to determine the effect of heating temperature on tensile strength, wear rate, and crystallinity of the magnesium/nano HA/shellac/cantala fiber bio-composite. This study used magnesium, nano-hydroxyapatite, shellac, and cantala fiber materials mixed using a blender with a volume ratio of magnesium/nano-HA-shellac/cantala fiber of 50/20/30, then compacted with a pressure of 300 MPa for ten minutes. The heating process was carried out with variations in temperature of 100 °C, 120 °C, 140 °C and 160 °C for two hours. The results showed that the lowest wear rate was 0.72 x 10^{-3} mm^3/Nm at a temperature variation of 160 °C. The highest tensile strength value was 6.58 MPa at 160 °C temperature variation. The highest degree of crystallinity, 74.15%, was obtained by observing X-ray diffraction (XRD) at a temperature variation of 160 °C.

1 Introduction

Bone fracture is a type of failure, bone dysfunction, or bone trauma that congenital disabilities, disease, or accidents can cause [1]. The problem of bone trauma that causes damage to parts of the bone is a challenge in itself for orthopedic experts because the healing process is still ineffective or even doesn't go well [2]. In modern orthopedic medicine, one way to heal cases of bone fractures is by using a support plate that is attached to the area around the broken bone using bone screws [1]. Bone screws generally use metal as the primary material because of its high mechanical properties, such as stainless steel and titanium [3]. The use of metal as a bone implant material is believed to cause inhibition of bone growth due to the higher density and modulus of elasticity of the material compared to human bone [4]. In this case, a material with properties like human bones is needed.

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Along with the times, the concept of active biomaterials emerged, namely biodegradable materials that can be absorbed by the body as long as they are attached to the body and no longer require surgery to remove [5]. Hydroxyapatite (HA) is a material commonly used in the medical field for the manufacture of implants because it has properties and composition similar to human bones and teeth [6]. Magnesium is one of the materials used to develop biomaterials to treat bone fracture wear. Magnesium is used because it has good mechanical properties and a fast rate of degradation in the body [7]. Shellac is used because it has properties that are easily degraded by the human body and is found in nature, so it is renewable [7]. The mechanical properties of the magnesium/hydroxyapatite/shellac mixture can be improved by adding cantala fiber. Cantala fiber is a natural fiber obtained from the leaf extraction process of the Agave Cantala Roxb [8].

The heating process, often referred to as thermal treatment or curing, plays a critical role in the final properties of the bio-composite, including its mechanical strength, biodegradability, and biocompatibility. However, the effects of different heating temperatures on these properties still need to be better understood. Current research has mainly focused on a limited range of temperatures, and more comprehensive studies need to evaluate a broader spectrum of heating temperatures. Furthermore, the influence of heating temperature on the microstructure and phase composition of bio-composites remains unclear. Addressing this gap will require systematic investigations to determine the optimal heating temperature that balances the desired mechanical and biological properties of bio-composite bone screws. In this study, Cantala fiber-reinforced Magnesium/HA/Shellac material was used for bone screw applications. The material that has been made is treated with heating temperatures of 100 °C, 120 °C, 140 °C, and 160 °C. The results of this study are expected to be an alternative material for making bone screws and are helpful for people with cracks or fractures.

2 Experimental Methods

2.1 Materials

The materials used in this study were Magnesium Powder, Hydroxyapatite Powder, shellac, and Cantala Fiber. Magnesium was used for matrices, HA was coated with Shellac and used as a filler to reduce porosity, and Cantala fibers acted as reinforcement in this biomaterial. The mechanical properties of Magnesium (Mg) and HA are shown in Tables 1 and 2.

Table 1. Mechanical properties of Mg [9]

Description	Value	Unit
Size of grain	0.06-0.3	mm
Melting point	650	°C
Density	1.738	gr/cm^3

Table 2. Mechanical properties of HA [10]

Description	Value	Unit
Size of grain	0.05-0.2	mm
Melting point	1100	°C
Density	3.076	gr/cm^3

2.2 Methods

The process of making specimens in this study began with manufacturing shellac solution. The preparation of the shellac solution in this study was first to destroy the secretion of the shellac tick with a mortar until smooth and then continue stirring with a blender until it became a powder. After turning into powder, it was mixed with 96% ethanol. The ratio of secretions of lac ticks to ethanol in this mixture was 1:8, and the mixing process was carried out for four hours using a magnetic stirrer. The shellac solution was then allowed to settle for a while, followed by filtration. The result of this filtration was a shellac solution. The HA/shellac mixing process was carried out using a magnetic stirrer at a temperature of 100

°C and a speed of 200 rpm for two hours. Then, mix the HA with shellac again with magnesium using a magnetic stirrer at 200 rpm. The magnesium mixed with HA shellac was remixed with cantala fiber in a 1:3 ratio in a mixer. This mixing process took three minutes. The mixed material was then fed into a tablet-shaped mold of 17 mm x 5 mm and a 10 mm x 10 mm cylinder. The mold was then placed on a press with a pressure of 300 MPa for forming. After the specimen compaction, it was formed into a green compact. Furthermore, the heating process was carried out with an oven machine with temperature variations of 100 °C, 120 °C, 140 °C, and 160 °C held for two hours for each temperature variation. This process aims to strengthen and harden the specimen. This specimen from the heating process was a degradable bone screw material. Tensile strength testing in this study used the Universal Testing Machine (UTM) using a 50 kg load cell, and specimens used American Society for Testing and Material (ASTM) D638 standards. Testing the wear rate in this study used a Pin-On-Disc Tribometer machine with parameters of 501.58 m track length, 1 m/s friction speed, and 2 kg loading according to ASTM G-99. Crystallinity analysis in this study used an X-ray diffraction (XRD) test kit and a diffraction scan angle between 10°-50°.

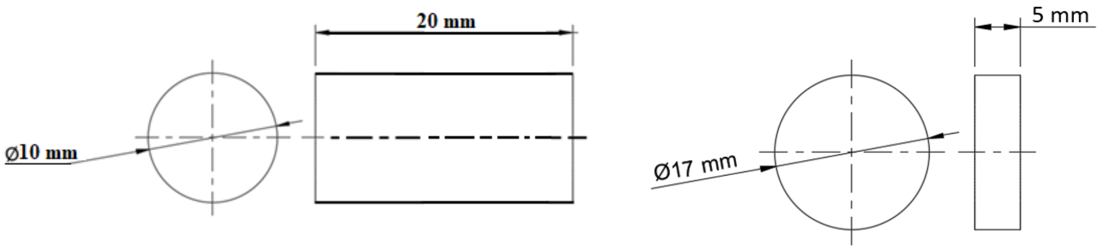

Figure 1. Dimension specimen test

3 Results and Discussion

3.1 Wear rate test

Figure 2 is the result of the wear rate test on the magnesium/HA/shellac/cantala fiber specimen, showing that the higher the heating temperature on the specimen, the lower the specific wear rate. The specific wear value at heating temperatures of 100 °C, 120 °C, 140 °C and 160 °C was 1.19 x 10^{-3} mm^3/Nm; 0.94 x 10^{-3} mm^3/Nm; 0.81 x 10^{-3} mm^3/Nm; 0.72 x 10^{-3} mm^3/Nm. The test results for the wear rate of the specimens were still above the wear rate of implants made from HA/shellac/cantala fiber, which was 0.51 x 10^{-3} mm^3/Nm [11] The wear rate for humans was 0.082 x 10^{-3} mm^3/Nm.

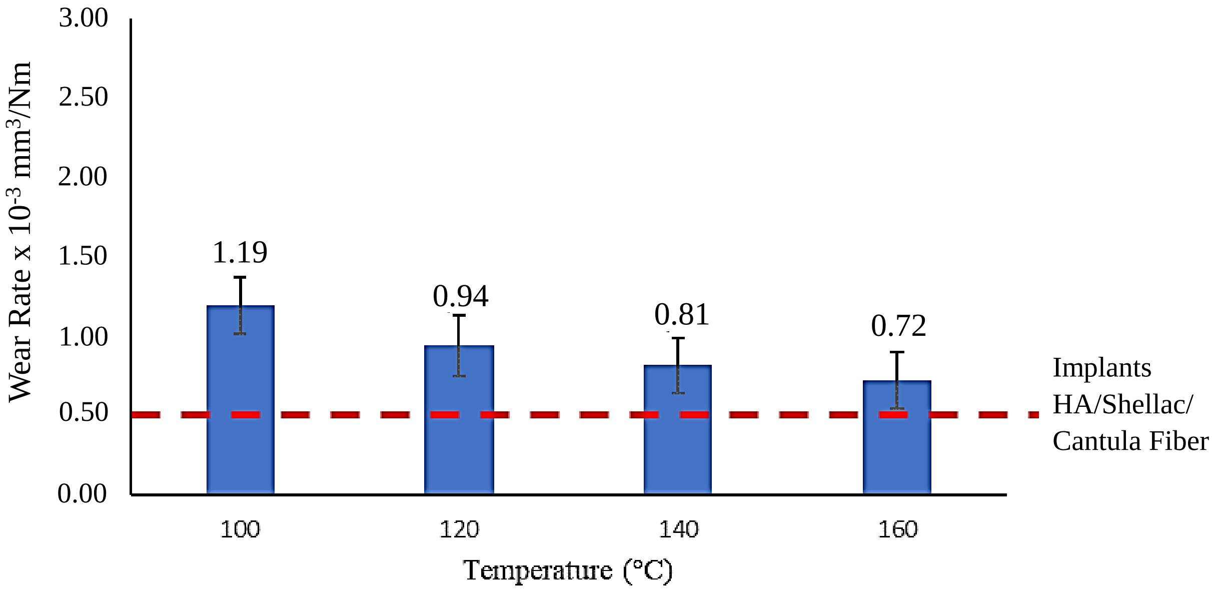

Figure 2. Wear rate test results

The lowest specific wear rate value on the magnesium/HA/shellac/cantala fiber specimen was found at a temperature of 160 °C, which was 0.72 x 10^{-3} mm^3/Nm. The results of the wear rate test showed that the value of the material wear rate decreased as the heating temperature increased; this is due to shellac having a melting point of 75-80 °C. Shellac, when heated at temperatures above 100 °C, will cause a change. Shellac, which was initially a rigid solid, became a viscous liquid [12].

3.2 Tensile strength test

Figure 3 shows the tensile test results; from the tensile test, the tensile strength value of the tested magnesium/HA/shellac/cantala fiber was obtained. From these data, it can be seen that the value of the material's tensile strength increased with the increase in heating temperature. The tensile strength values at 100, 120, 140, and 160 °C were 4.8, 4.85, 5.9, and 6.58 MPa. The results of the tensile strength of the magnesium/HA/shellac/cantala fiber specimens when compared with the tensile strength value of human bone, which was 2.54 MPa [13], the tensile strength value was above the tensile strength value of human bone when compared with the tensile strength value in stainless material steel, which is 465 MPa [14], the tensile strength value of the magnesium/HA/shellac/cantala fiber specimen was still below the strength value of stainless steel.

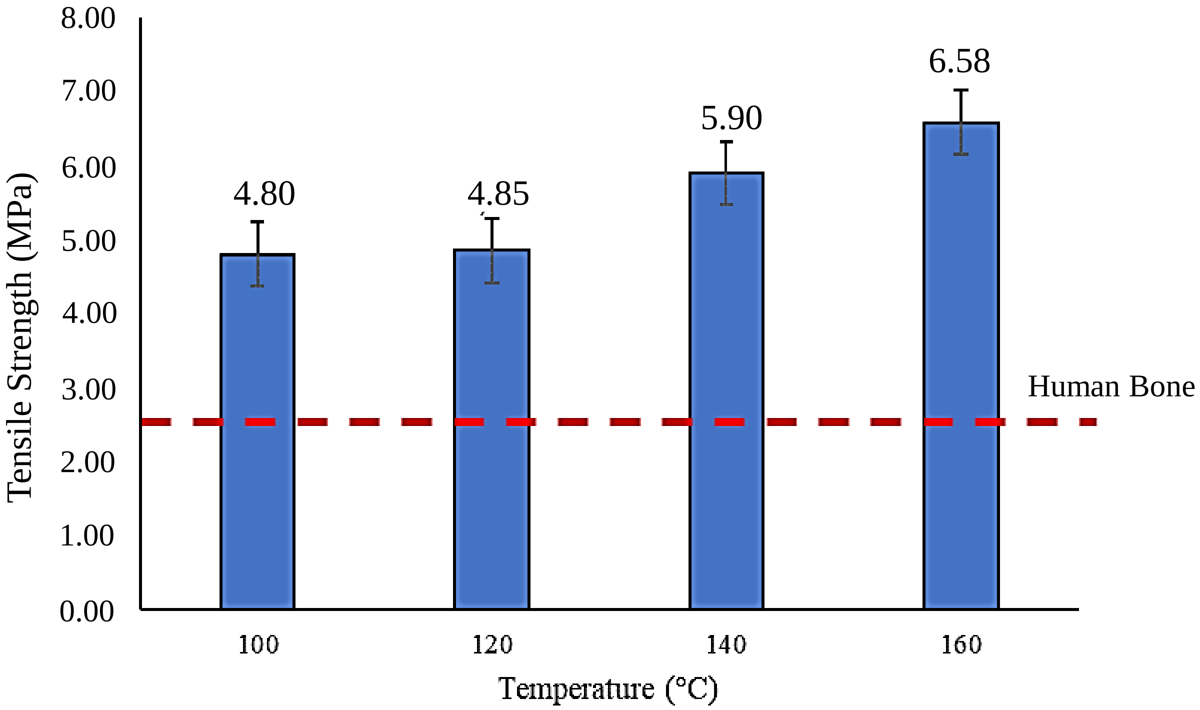

Figure 3. Tensile strength test results

The tensile strength of magnesium/HA/shellac/cantala Fiber increased with increasing temperature, with the highest tensile strength at 160 °C of 6.58 MPa. This is because the higher the heating temperature on the specimen, the stronger the bond between the powders on the specimen will increase. Where the magnesium powder in the specimen will be denser with increasing heating temperature so that the magnesium powder will strengthen the bond with other powders contained in the specimen [15].

3.3 Crystallinity

Table 3 shows that the degree of crystallinity of magnesium/HA/shellac/cantala fiber increased with the heating temperature applied to the specimen. The lowest degree of crystallinity was found at a heating temperature of 100 °C which was 50.60%, then at 120 °C, it was 67.80%; at 140 °C, it was 67.81%; and the highest was at a heating temperature of 160 °C with a degree of crystallinity of 74.15%.

Table 3. Crystallinity of magnesium/HA/shellac/cantala fiber

Temperature (°C)	Crystallinity (%)
100	50.6
120	67.8
140	67.81
160	74.15

The degree of crystallinity is a value that states the amount of crystal intensity in a material. In the magnesium/HA/shellac/cantala fiber specimens that XRD has observed, variations in heating temperature affect the number of crystal intensities in the specimen. The higher the heating temperature, the higher the degree of crystallinity of the specimen. This is because at temperatures below 110 °C, little hydroxyapatite synthesis is formed [16], and the higher the heating temperature in the specimen, the more crystals are formed because the arrangement of atoms in the specimen is more regular [17].

4 Conclusions

Based on the testing process, observations, and the results of discussions carried out in the study, conclusions can be drawn. The wear rate test of magnesium/HA/shellac/cantala specimens had the lowest wear rate value at a heating temperature of 160 °C, which was 0.72 x 10^{-3} mm^3/Nm, while magnesium/HA/shellac/cantala specimens had the highest wear rate value at a heating temperature of 100°C of 1.19 x 10^{-3} mm^3/Nm. Magnesium/HA/shellac/cantala specimen tensile strength testing had the highest value at a heating temperature of 160 °C of 6.58 MPa. The magnesium/HA/shellac/cantala specimen had the lowest tensile strength value at a heating temperature of 100 °C of 4.8 MPa. The crystallinity of magnesium/HA/shellac/cantala fiber increased with the heating temperature, with the highest crystallinity degree at a heating temperature of 160 °C being 74.15%, while the lowest crystallinity degree at a heating temperature of 100 °C was 50.6%. Future research on the heating temperature of bio-composites for bone screws should focus on conducting systematic studies to investigate a wide range of temperatures and their effects on the material properties. This should comprehensively characterize the bio-composite's mechanical strength, biodegradability, and biocompatibility at different heating temperatures. Additionally, studies should aim to understand the influence of heating temperature on the microstructure and phase composition of the bio-composite. Developing predictive models based on these findings could help in determining the optimal heating temperature for achieving the desired properties of bio-composite bone screws. Moreover, investigating novel heating techniques, such as microwave or infrared heating, and their effects on bio-composite properties could also be valuable for future research.

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